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Chemical Analyses – Determination of loss on ignition in sediment, sludge, soil, and waste

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Foreword

This document is developed in the project 'Horizontal'. It is a result of a desk study on dry matter, loss on ignition and total organic carbon in the project that aims at describing horizontal standards to be used for sludge, soil and biowaste. After discussion with all parties concerned in CEN the standard will be developed further as a modular horizontal method and validated in the project 'Horizontal'.

Until now test methods determining properties of materials were often prepared in Technical Committees (TC's) working on specific products or specific sectors. In the growing amount of product and sector oriented test methods it was recognised that many steps in test procedures are or could be used in test procedures for many products, materials and sectors. It was supposed that, by careful determination of these steps and selection of specific questions within these steps, elements of the test procedure could be described in a way that can be used for all materials and products or for all materials and products with certain specifications.

Based on this hypothesis a horizontal modular approach is being investigated and developed in the project 'Horizontal'. 'Horizontal' means that the methods can be used for a wide range of materials and products with certain properties. 'Modular' means that a test standard developed in this approach concerns a specific step in a test procedure and not the whole test procedure (from sampling to analyses).

The use of modular horizontal standards implies the drawing of test schemes as well. Before executing a test on a certain material or product to determine certain characteristics it is necessary to draw up a protocol in which the adequate modules are selected, together forming the basis for the test procedure.

This standard is a module for determination of loss on ignition of dry mass. This module concerns the determination of loss on ignition after the dry matter has been determined in accordance with the method of EN XXXXX:200X.

The other horizontal modules that will be available in due time are to be found in the informative annex [*xxx*] which contains a brief overview of the modules that are or will be worked out in the project 'Horizontal.'

The texts of chapters 1 to 11 are normative; annexes are informative, as stated in the top lines of the annexes.

1 Scope

This European Standard specifies a method for the determination of the loss on ignition of dry mass of sludge, sediment, soil, and waste at 550ºC after the dry matter has been determined in accordance with the method of EN XXXXX:200X.

This method applies to the determination of loss on ignition of:

- sludges, including liquid, paste-like or solid sludges
- all types of soil samples
- sediments, and
- waste.

NOTE The loss on ignition is often used as an estimate ofr the content of non-volatile organic matter in the sample. It should be noted that inorganic substances or decomposition products (e.g. H_2O, CO_2, SO_2, O_2) are released or absorbed and some inorganic substances are volatile under the reaction conditions.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references, the latest edition of the publication referred to applies (including amendments).

EN XXXXX: 200X.Determination of dry matter in sediment, sludge, soil, and waste – gravimetric method

EN XXXXX: 200X. Sample preparation

3 Terms and definitions

For the purpose of this European Standard, the following terms and definitions apply.

3.1 Loss on ignition (LOI)

The change in mass as a result of heating a sample under specified conditions. The loss on ignition (LOI) is expressed as a weight percentage of the dry mass.

3.2 Residue on ignition

The mass remaining after heating a sample under specified conditions. The residue on ignition is related to the dry mass and expressed in percentage.

3.3 Dry mass

The mass of sample obtained after the specified drying process. It is expressed in grams or kilograms.

3.4 Dry matter, w_{dm}

Dry residue after drying according to the specified drying process. It is expressed as a percentage or in grams per kilogram.

3.5 Constant mass

Constant mass is reached when, during the ignition process, the difference between two successive weighings of the sample at an interval of 1 hour, first heated, then cooled to room temperature, is within 0,5% (m/m) of the last determined mass or 2 mg, whichever is the greater.

Note 2 Usually 2 h are sufficient for most soils, sludges, waste and biowaste samples, but certain sample types and large samples require longer drying periods.

4 Principle

A dried test sample is heated in a furnace to constant mass at $(550 \pm 25)^{o}C$.

The difference in mass before and after the ignition process is used to calculate the loss on ignition.

5 Interferences

As LOI is an empirical parameter, in principle there is no interference connected to the determination. However, for many purposes the determination of LOI is used for the assessment of the content of organic matter in the sample. In this case, the loss of volatile inorganic substances can occur giving high results.

Note 1 Chemically bound water could be released during heating, thereby contributing to the loss on ignition.

Note 2 Iron or other metals present in the sample in metallic state could be oxidised during heating, thereby producing lower results.

Note 3 Sulphides present in the sample could be oxidised to sulphate during heating, thereby producing lower results.

Note 4 Explosive ignition is likely to result in loss of residue from the crucible, thereby contributing to the loss on ignition.

Note 5 Calcium hydroxide or calcium oxide present in large amounts (e.g. sludge conditioned with lime) may combine with sulphuric oxides liberated during ignition or with carbon dioxide formed during ignition producing lower results.

6 Storage

During storage, samples may be subject to changes (e.g. uptake or liberation of water, carbon dioxide and other volatiles), which are liable to falsify the results. Biological active samples should be analysed within 3 days. If analysed within this period, the samples should be stored at about 4^{o}C; or otherwise stored directly at maximum - 18^{o}C. Other samples may be stored in a closed container in a well-ventilated place.

7 Hazards

Samples of sludge, biowaste or contaminated soil are liable to ferment and usually contain harmful microorganisms. It is essential to keep them away from any food or drink, and to protect any skincuts. Bursting bottles containing e.g. sludge can produce microorganism-contaminated shrapnel and/or infectious aerosols.

When handling sludge and biowaste samples, it is necessary to wear gloves, face and eye protection, and sufficient body protection to guard against bottles bursting. Gasses evolved may be flammable.

Special precaution must be taken during the ignition process to prevent contamination of the laboratory atmosphere by flammable, explosive or toxic gasses.

8 Apparatus

8.1 Flat bottom crucible
(50 to 70) mm in diameter, suitable for ignition at 550^{o}C, e.g. made of nickel, porcelain, silica, or platinum.

8.2 Muffle furnace
or equivalent equipment, capable of maintaining a temperature of (550 $\pm$ 25) ^{o}C.

8.3 Desiccator
with active drying agent, such as silica gel.

8.4 Analytical balance
with an accuracy of 1 mg or better.

9 Procedure

9.1 Sludge, sediment, soil, and waste with low content of volatiles

If the determination of loss on ignition is carried out in the same crucible as the determination of dry residue, refer to EN XXXXX for the initial crucible weighing. If not, the sample is a representative portion of the dry mass obtained according to EN XXXXX:200X. Every necessary precaution shall be taken to avoid absorption of atmospheric humidity by the dried sample until weighed.

Place a crucible (8.1) in the furnace (8.2) and heat at (550 ± 25)°C for at least 30 min. After cooling in the desiccator (8.3) to ambient temperature, weigh to the nearest 1 mg, (m_a).

Weigh into the crucible 0.5 g to 5 g of the dried sludge to the nearest 1 mg, (m_b), and heat in the furnace (8.2) at (550 ± 25)°C for at least 60 min.

Note 1 If the dry mass has a high organic matter content, losses may occur as a result of rapid ignition or deflagration of the sample. In this case heat the sample slowly until ignition.

Place the hot crucible containing the residue on ignition in the desiccator and leave to cool.

After cooling in the desiccator to ambient temperature, weigh the crucible containing the dry residue to the nearest 1 mg (m_c).

The crucible is weighed immediately after removal from the desiccator and the weighing operation is completed as quickly as possible. The mass of the residue on ignition and thus the loss on ignition shall be regarded as constant if the mass obtained after a further half-hour period of ignition at 550°C in the preheated furnace, (m_c - m_a), differs max. 0.5% of the previous value or 2 mg, whichever is the greater (3.4).

Note 2 In cases when even after the third heating to 550°C constant mass is not obtained, record the value determined as the last measurement. The lack of constant mass should be reported together with the result.

9.2 Samples containing volatile substances

For samples containing significant amounts of volatile substances the dry matter cannot be determined as dry residue. In this case the dry matter shall be calculated from the water content. For procedure, see EN 14346 – Characterisation of waste – Calculation of dry matter by determination of dry residue or water content. In this case, the loss on ignition is performed directly on the un-dried sample.

Place a crucible (8.1) in the furnace (8.2) and heat at (550 ± 25)°C for at least 30 min. After cooling in the desiccator (8.3) to ambient temperature, weigh to the nearest 1 mg, (m_a).

Weigh into the crucible 0.5 g to 5 g of the sample to the nearest 1 mg, (m_b), and heat in the furnace (8.2) at (550 ± 25)°C for at least 60 min. Larger masses may be taken if appropriate. All necessary precautions should be taken to avoid absorption of atmospheric humidity or loss of volatiles from the samples until it has been weighed.

Note 1 To avoid splashing from escaping vapours or sudden fire it is recommended carefully stripping most of the volatile components from the sample in a drying oven or at ambient temperature in a fume hood prior to ignition.

Note 2 Samples containing highly flammable components e.g. solvents or waste oil should be ignited and allowed to burn in a fume hood before being inserted into the furnace.

Place the hot crucible containing the residue on ignition in the desiccator and leave to cool to room temperature. Weigh the crucible containing the dry residue to the nearest 1 mg (m_c).

Weighing is carried out immediately after removal of the crucible from the desiccator and the weighing operation is completed as quickly as possible. The mass of the residue on ignition - and therefore the loss on

ignition - shall be regarded as constant, if the mass obtained after a further half-hour period of ignition at 550°C in the pre-heated furnace, (m_c - m_a), does not differ by more than 0.5% of the previous value or 2 mg, whichever is the greater (3.4).

Note 3 In cases when even after the third heating to 550°C constant mass is not obtained, record the value determined as the last measurement. The lack of constant mass should be reported together with the result.

10 Expression of results

10.1 Calculation

The loss on ignition of the dry mass of a solid sample expressed in percentage shall be calculated from equation (1):

$$w_V = \frac{(m_b - m_c)}{(m_b - m_a)} \times 100$$

The residue on ignition of the dry mass of a solid sample expressed in percentage shall be calculated from equation (2):

$$W_R = 100 - w_V$$

where

w_V is the loss on ignition of the dry mass of a solid sample, in percentages;

W_R is the residue on ignition of the dry mass of a solid sample, in percentages;

m_a is the mass of the empty crucible, in grams;

m_b is the mass of the crucible containing the dry mass, in grams;

m_c is the mass of the crucible containing the ignited dry mass, in grams;

The results shall be rounded to the nearest 0.1%.

10.2 Precision data

See Annex A:

11 Test report

The test report shall contain the following information:

a) Reference to this European Standard;

b) Reference to the standard used for determination of dry matter or water content;

c) All information necessary for complete identification of the sludge sample;

d) Results of the determination according to clause 10;

e) Any details not specified in this European Standard and any other factor that may have affected the results.

Annex A

Performance data of the interlaboratory comparison

(Informative)

Performance data of the interlaboratory comparison from EN 12879 can be used for sludge samples, provided description of samples are provided

Bibliography